

Date : August 15, 2019

CERTIFICATE OF ANALYSIS – GC PROFILING

SAMPLE IDENTIFICATION

Internal code : 19H08-PTH02-1-JSB

Customer identification : Black Pepper - India - B4010691R

Type : Essential oil

Source : *Piper nigrum*

Customer : Plant Therapy

ANALYSIS

Method: PC-PA-014 - Analysis of the composition of an essential oil, or other volatile liquid, by FAST GC-FID (in French); identifications validated by GC-MS.

Analyst : Lindsay Girard, B. Sc.

Analysis date : August 13, 2019

Checked and approved by :

Sylvain Mercier, M. Sc., chimiste 2014-005

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PHYSICOCHEMICAL DATA

Physical aspect: Faintly yellow liquid

Refractive index: 1.4842 ± 0.0003 (20 °C)

ISO 3061:2008 - OIL OF BLACK PEPPER - INDIA

Compound	Min. %	Max. %	Observed %	Complies?
Caryophyllene oxide		1.0	0.7	Yes
α-Selinene		3.0	0.2	Yes
β-Selinene	0.5	3.5	0.4	No
Germacrene D		2.0	0.5	Yes
β-Caryophyllene	12	29	27	Yes
α-Copaene	0.5	4.5	3.7	Yes
δ-Elemene	0.5	3.5	1.9	Yes
Limonene	10	17	10	Yes
Δ ³ -Carene	3	15	4	Yes
Sabinene	6	15	12	Yes
β-Pinene	5	12	9	Yes
α-Pinene	3	12	11	Yes
Refractive index	1.4780	1.4870	1.4842	Yes

CONCLUSION

No adulterant, contaminant or diluent has been detected using this method. The oil marginally does not comply with the ISO norm for black pepper oil.

ANALYSIS SUMMARY – CONSOLIDATED CONTENTS

New readers of similar reports are encouraged to read table footnotes at least once.

Identification	%	Classe
Isovaleral	tr	Aliphatic aldehyde
Toluene	tr	Simple phenolic
Tricyclene	0.03	Monoterpene
α -Thujene	0.91	Monoterpene
α -Pinene	10.84	Monoterpene
Camphene	0.29	Monoterpene
meta-Cymene	0.01	Monoterpene
β -Pinene	9.11	Monoterpene
Sabinene	11.70	Monoterpene
Dehydro-1,8-cineole	0.01	Monoterpenic ether
Myrcene	1.21	Monoterpene
α -Phellandrene	0.74	Monoterpene
Pseudolimonene	0.05	Monoterpene
Δ^3 -Carene	3.79	Monoterpene
1,4-Cineole	0.16*	Monoterpenic ether
α -Terpinene	[0.16]*	Monoterpene
para-Cymene	0.53	Monoterpene
Limonene	9.60	Monoterpene
1,8-Cineole	0.92*	Monoterpenic ether
β -Phellandrene	[0.92]*	Monoterpene
(Z)- β -Ocimene	0.02	Monoterpene
(E)- β -Ocimene	0.11	Monoterpene
γ -Terpinene	0.30	Monoterpene
cis-Sabinene hydrate	0.08	Monoterpenic alcohol
cis-Linalool oxide (fur.)	0.01	Monoterpenic alcohol
Isoterpinolene	0.05	Monoterpene
Terpinolene	0.32	Monoterpene
para-Cymenene	0.02	Monoterpene
trans-Sabinene hydrate	0.08	Monoterpenic alcohol
Unknown	0.02	Unknown
Linalool	0.27	Monoterpenic alcohol
trans-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
cis-Limonene oxide	0.04	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.02	Monoterpenic alcohol
trans-Verbenol	0.02	Monoterpenic alcohol
meta-Mentha-4,6-dien-8-ol	0.02	Monoterpenic alcohol
Pinocarvone	0.01	Monoterpenic ketone
cis-Sabinol	0.01	Monoterpenic alcohol
Terpinen-4-ol	0.56	Monoterpenic alcohol
meta-Cymen-8-ol	0.03	Monoterpenic alcohol
para-Cymen-8-ol	0.03	Monoterpenic alcohol
Methyl salicylate	0.01	Phenolic ester
α -Terpineol	0.09	Monoterpenic alcohol
Myrtenol	0.04	Monoterpenic alcohol
α -Phellandrene epoxide	0.02	Monoterpenic ether
Unknown	0.01	Oxygenated monoterpene
Verbenone	0.01	Monoterpenic ketone

Car-2-en-4-one?	0.02	Monoterpenic ketone
<i>trans</i> -Carveol	0.01	Monoterpenic alcohol
<i>cis</i> -Carveol	0.02	Monoterpenic alcohol
Carvone	0.01	Monoterpenic ketone
Unknown	0.04	Unknown
Methyl citronellate	0.03	Monoterpenic ester
<i>trans</i> -Ascaridole glycol	0.03	Monoterpenic alcohol
Unknown	0.01	Monoterpenic ester
Unknown	0.01	Oxygenated monoterpene
Unknown	0.02	Unknown
Unknown	0.01	Unknown
Myrtenyl acetate	0.01	Monoterpenic ester
δ -Elemene	1.93	Sesquiterpene
Bicycloelemene	0.01	Sesquiterpene
α -Cubebene	0.27	Sesquiterpene
Cyclosativene I	0.10	Sesquiterpene
Cyclosativene II	0.03	Sesquiterpene
α -Copaene	3.66	Sesquiterpene
<i>cis</i> - β -Elemene	0.05	Sesquiterpene
β -Cubebene	0.38	Sesquiterpene
β -Elemene	0.38	Sesquiterpene
Isocaryophyllene	0.08	Sesquiterpene
α -Gurjunene	0.11	Sesquiterpene
β -Caryophyllene	27.22	Sesquiterpene
β -Copaene	0.21	Sesquiterpene
α -Guaiene	0.13*	Sesquiterpene
<i>trans</i> - α -Bergamotene	[0.13]*	Sesquiterpene
Unknown	0.02	Unknown
Unknown	0.01	Sesquiterpene
α -Humulene	1.73	Sesquiterpene
allo-Aromadendrene	0.04	Sesquiterpene
(<i>E</i>)- β -Farnesene	0.15	Sesquiterpene
β -Santalene	0.04	Sesquiterpene
γ -Muurolene	0.17	Sesquiterpene
Germacrene D	0.46	Sesquiterpene
α -Curcumene	0.19	Sesquiterpene
β -Selinene	0.35	Sesquiterpene
<i>trans</i> -Muurola-4(15),5-diene	0.12	Sesquiterpene
Viridiflorene	0.11	Sesquiterpene
epi-Cubebol	0.21	Sesquiterpenic alcohol
α -Selinene	0.24	Sesquiterpene
Bicyclogermacrene	0.10	Sesquiterpene
α -Muurolene	0.69	Sesquiterpene
β -Bisabolene	2.54	Sesquiterpene
7-epi- α -Selinene	0.02	Sesquiterpene
Cubebol	0.28	Sesquiterpenic alcohol
<i>trans</i> -Calamenene	0.15	Sesquiterpene
δ -Cadinene	1.89	Sesquiterpene
α -Calacorene	0.06	Sesquiterpene
(<i>E</i>)- α -Bisabolene	0.16	Sesquiterpene
Isocaryophyllene epoxide B	0.16	Sesquiterpenic ether
Germacrene B	0.07	Sesquiterpene

(E)-Nerolidol	0.16	Sesquiterpenic alcohol
Spathulenol	0.05	Sesquiterpenic alcohol
Caryophyllene oxide	0.66	Sesquiterpenic ether
Caryophyllene oxide isomer	0.14	Sesquiterpenic ether
Unknown	0.01	Oxygenated sesquiterpene
Humulene epoxide I	0.01	Sesquiterpenic ether
Humulene epoxide II	0.04	Sesquiterpenic ether
α -Corocalene	0.02	Sesquiterpene
Guaia-6,10(14)-dien-4 β -ol	0.16	Sesquiterpenic alcohol
Caryophylladienol I	0.02	Sesquiterpenic alcohol
Caryophylladienol II	0.03	Sesquiterpenic alcohol
τ -Cadinol	0.02	Sesquiterpenic alcohol
τ -Muurolol	0.08	Sesquiterpenic alcohol
α -Muurolol	0.28	Sesquiterpenic alcohol
<i>cis</i> -Calamene-10-ol	0.03	Sesquiterpenic alcohol
<i>trans</i> -Calamene-10-ol	0.02	Sesquiterpenic alcohol
(3Z)-Caryophylla-3,8(13)-dien-5 β -ol	0.02	Sesquiterpenic alcohol
Unknown	0.01	Oxygenated sesquiterpene
Consolidated total	98.67%	

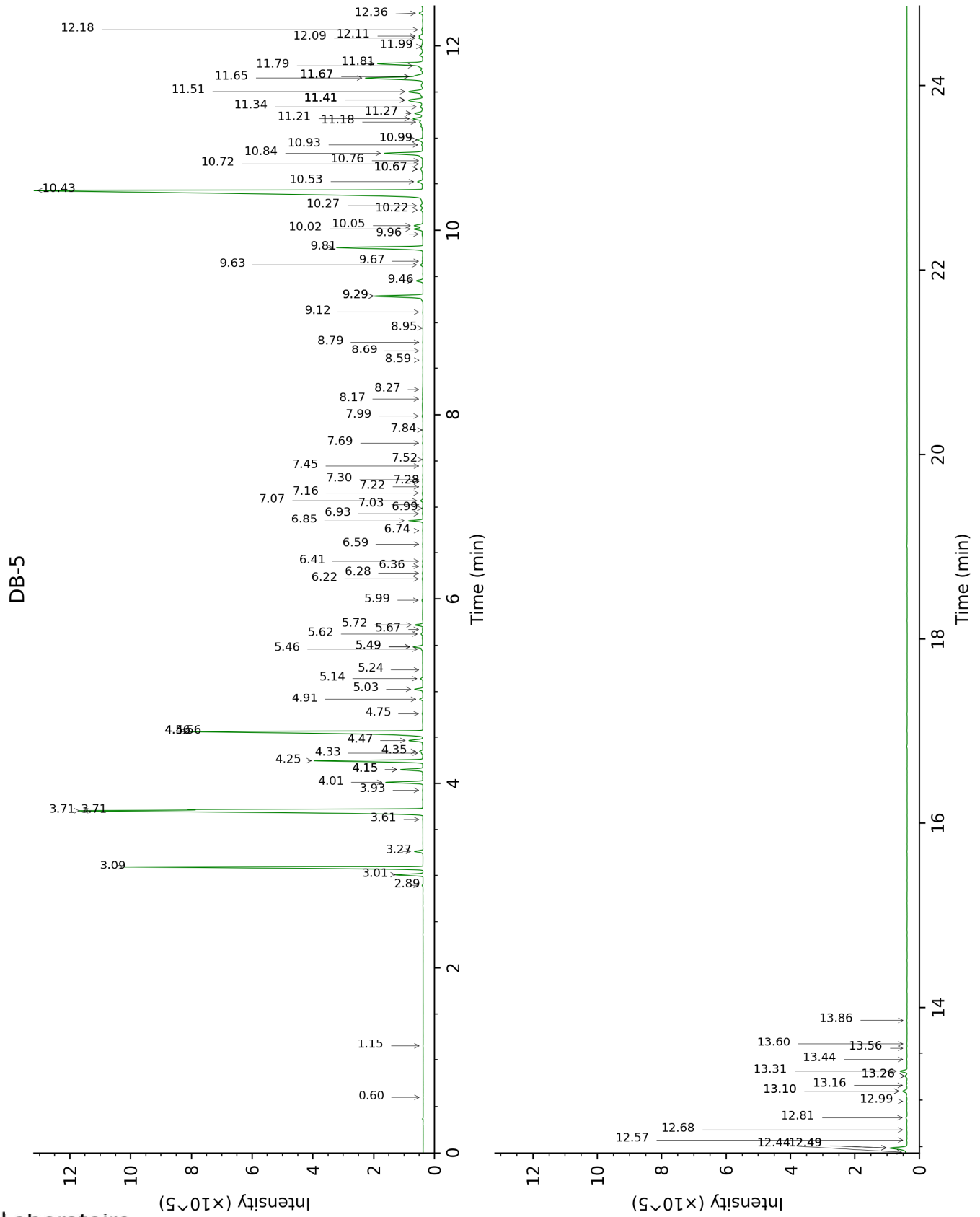
*: Individual compounds concentration could not be found due to overlapping coelutions on columns considered
[xx]: Duplicate percentage due to coelutions, not taken into account in the consolidated total
tr: The compound has been detected below 0.005% of total signal.

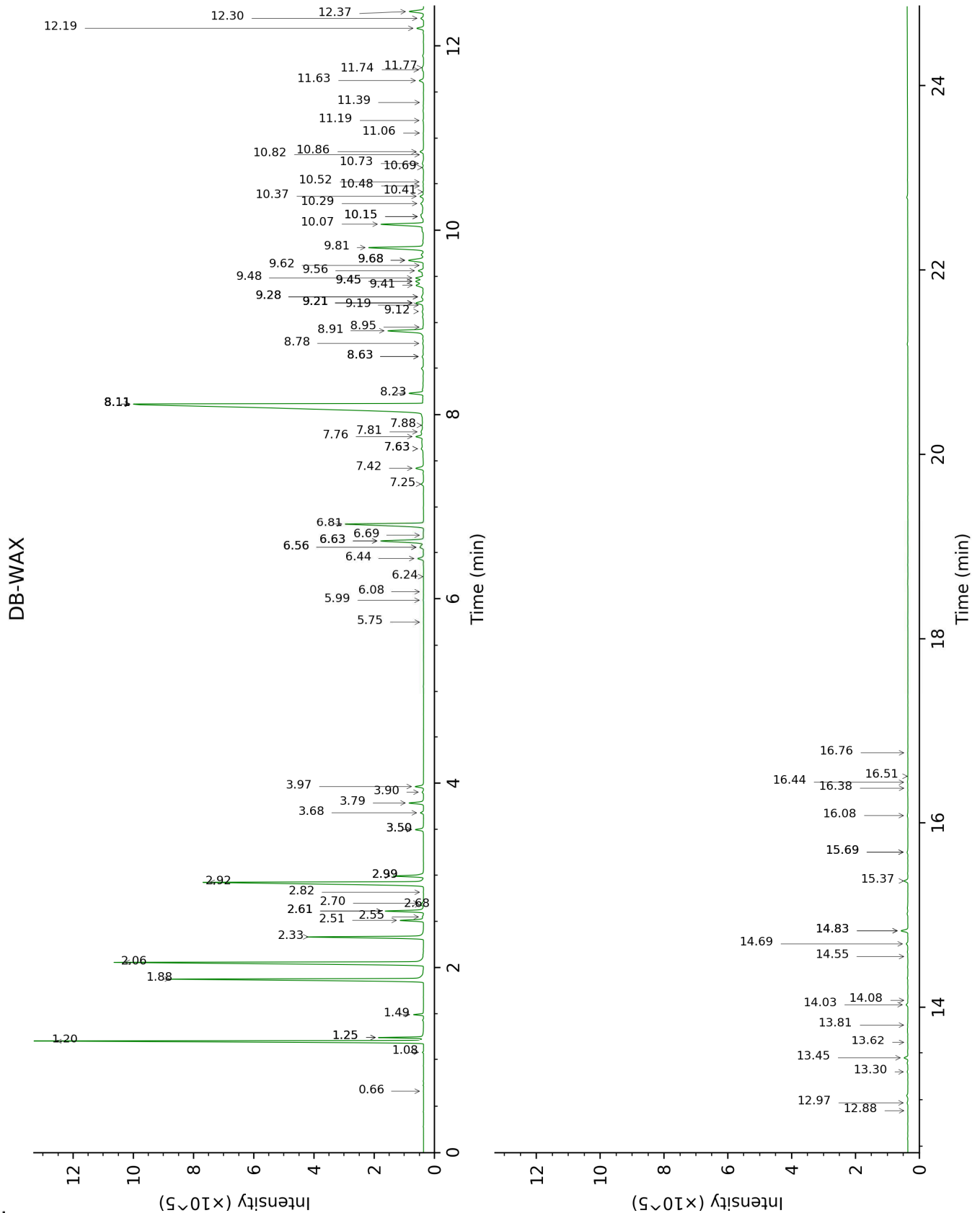
Note: no correction factor was applied

About "consolidated" data: The table above presents the breakdown of the sample volatile constituents after applying an algorithm to collapse data acquired from the multi-columns system of PhytoChemia into a single set of consolidated contents. In case of discrepancies between columns, the algorithm is set to prioritize data from the most standard DB-5 column, and smallest values so as to avoid overestimating individual content. This process is semi-automatic. Advanced users are invited to consult the "Full analysis data" table after the chromatograms in this report to access the full untreated data and perform their own calculations if needed.

Unknowns: Unknown compounds' mass spectral data is presented in the "Full analysis data" table. The occurrence of unknown compounds is to be expected in many samples, and does not denote particular problems unless noted otherwise in the conclusion.

This page was intentionally left blank. The following pages present the complete data of the analysis.





FULL ANALYSIS DATA

Identification	Column DB-5			Column DB-WAX		
	R.T	R.I	%	R.T	R.I	%
Isovaleral	0.60	640	tr	0.66	887	tr
Toluene	1.15	760	tr	1.25*	1000	1.01
Tricyclene	2.89	918	0.03	1.08	970	0.03
α -Thujene	3.01	925	0.91	1.25*	1000	[1.01]
α -Pinene	3.09	931	10.84	1.20	993	10.94
Camphene	3.27	942	0.29	1.49	1026	0.25
meta-Cymene	3.61	965	0.01	2.61*	1134	1.24
β -Pinene	3.71*	971	20.53	1.88	1066	9.11
Sabinene	3.71*	971	[20.53]	2.06	1085	11.70
Dehydro-1,8-cineole	3.93	986	0.01	2.82	1150	0.01
Myrcene	4.01	992	1.21	2.61*	1134	[1.24]
α -Phellandrene	4.15*	1001	0.78	2.51	1125	0.74
Pseudolimonene	4.15*	1001	[0.78]	2.55	1128	0.05
Δ^3 -Carene	4.25	1007	3.79	2.33	1111	3.83
1,4-Cineole	4.33†	1012	0.16	2.70†	1140	[0.16]
α -Terpinene	4.35†	1013	[0.16]	2.68†	1139	0.16
para-Cymene	4.47	1020	0.53	3.79	1227	0.56
Limonene	4.56*	1026	10.53	2.92	1159	9.60
1,8-Cineole	4.56*	1026	[10.53]	2.99*	1164	1.04
β -Phellandrene	4.56*	1026	[10.53]	2.99*	1164	[1.04]
(Z)- β -Ocimene	4.76	1039	0.02	3.50*	1205	0.34
(E)- β -Ocimene	4.91	1048	0.11	3.68	1219	0.12
γ -Terpinene	5.03	1056	0.30	3.50*	1205	[0.34]
cis-Sabinene hydrate	5.14	1063	0.08	6.56*	1429	0.24
cis-Linalool oxide (fur.)	5.24	1069	0.01	6.24	1405	0.01
Isoterpinolene	5.46	1083	0.05	3.90	1236	0.06
Terpinolene	5.49*	1085	0.35	3.97	1240	0.32
para-Cymenene	5.49*	1085	[0.35]	5.99	1387	0.02
trans-Sabinene hydrate	5.62	1094	0.08	7.63*	1510	0.08
Unknown [m/z 109, 43 (65), 95 (54), 119 (50), 91 (47)... 149 (8)...]	5.67	1097	0.02	5.75	1370	0.01
Linalool	5.72	1100	0.27	7.76	1520	0.31
trans-para-Mentha-2,8-dien-1-ol	5.99	1117	0.03	8.63*	1587	0.06
cis-Limonene oxide	6.22	1132	0.04	6.08	1393	0.01
cis-para-Mentha-2,8-dien-1-ol	6.28	1136	0.02	9.12	1627	0.07
trans-Verbenol	6.36	1141	0.02	9.19	1632	0.04
meta-Mentha-4,6-dien-8-ol	6.41	1144	0.02	8.95	1613	0.01

Pinocarvone	6.59	1156	0.01	7.63*	1510	[0.08]
<i>cis</i> -Sabinol	6.74	1166	0.01	10.48	1739	0.01
Terpinen-4-ol	6.85	1173	0.56	8.23	1556	0.53
meta-Cymen-8-ol	6.93	1179	0.03			
para-Cymen-8-ol	6.99	1182	0.03	11.19	1800	0.03
Methyl salicylate	7.03	1185	0.01	10.15*†	1711	0.24
α-Terpineol	7.07	1188	0.09	9.45*	1653	0.42
Myrtenol	7.16	1193	0.04	10.52	1742	0.02
α-Phellandrene epoxide	7.22	1198	0.02	10.69	1756	0.05
Unknown [m/z 109, 91 (100), 81 (88), 94 (75), 119 (74), 96 (73), 41 (63)... 150 (2)]	7.28	1202	0.01	10.42	1733	0.02
Verbenone	7.30	1203	0.01	9.28*	1640	0.13
Car-2-en-4-one?	7.45	1213	0.02	9.22*	1634	0.35
<i>trans</i> -Carveol	7.52	1218	0.01	11.06	1788	0.01
<i>cis</i> -Carveol	7.69	1230	0.02	11.39	1817	0.01
Carvone	7.84	1240	0.01	9.62	1667	0.02
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.99	1250	0.04	10.82	1768	0.01
Methyl citronellate	8.17	1263	0.03	7.88	1529	0.03
<i>trans</i> -Ascaridole glycol	8.27	1270	0.03	13.81	2039	0.03
Unknown [m/z 93, 43 (60), 108 (58), 69 (36), 41 (35)... 150 (5), 184 (1)]	8.59	1292	0.01			
Unknown [m/z 43, 93 (66), 91 (44), 41 (38), 69 (35)... 152? (1)]	8.69	1299	0.01			
Unknown [m/z 43, 111 (84), 109 (71), 126 (70)...]	8.79	1306	0.02	14.08	2065	0.01
Unknown [m/z 111, 126 (93), 43 (90), 71 (60)...]	8.94	1311	0.01	14.83*	2139	0.28
Myrtenyl acetate	9.12	1324	0.01	9.22*	1634	[0.35]
δ-Elemene	9.29*	1336	1.98	6.63*	1434	1.96
Bicycloelemene	9.29*	1336	[1.98]	6.69	1439	0.01
α-Cubebene	9.46	1348	0.27	6.44	1420	0.27
Cyclosativene I	9.63	1360	0.10	6.56*	1429	[0.24]
Cyclosativene II	9.67	1362	0.03	6.63*	1434	[1.96]
α-Copaene	9.82	1373	3.66	6.81	1448	3.62
<i>cis</i> -β-Elemene	9.96	1383	0.05	8.11*	1547	27.09
β-Cubebene	10.02	1387	0.38	7.42	1493	0.35
β-Elemene	10.05	1390	0.38	8.11*	1547	[27.09]
Isocaryophyllene	10.22	1402	0.08	7.81	1524	0.10

α -Gurjunene	10.27	1405	0.11	7.25	1481	0.10
β -Caryophyllene	10.43	1417	27.22	8.11*	1547	[27.09]
β -Copaene	10.53	1424	0.21	8.11*	1547	[27.09]
α -Guaiene	10.67*	1435	0.13	8.11*	1547	[27.09]
<i>trans</i> - α - Bergamotene	10.67*	1435	[0.13]	8.11*	1547	[27.09]
Unknown [m/z 41, 97 (78), 69 (77), 43 (71), 125 (67), 55 (56)... 168 (39)]	10.72	1439	0.02	16.76	2340	0.01
Unknown [m/z 139, 69 (60), 41 (51), 43 (47), 119 (41)... 204 (1)]	10.76	1442	0.01			
α -Humulene	10.84	1448	1.73	8.91	1610	1.66
allo- Aromadendrene	10.93	1455	0.04	8.63*	1587	[0.06]
(<i>E</i>)- β -Farnesene	10.99*	1459	0.25	9.22*	1634	[0.35]
β -Santalene	10.99*	1459	[0.25]	8.78	1599	0.04
γ -Muuroolene	11.18	1473	0.17	9.22*	1634	[0.35]
Germacrene D	11.21	1476	0.46	9.41	1650	0.42
ar-Curcumene	11.27*	1480	0.38	10.29	1723	0.19
β -Selinene	11.27*	1480	[0.38]	9.48	1656	0.35
<i>trans</i> -Muurolo- 4(15),5-diene	11.34	1485	0.12	9.45*	1653	[0.42]
Viridiflorene	11.42*	1491	0.85	9.28*	1640	[0.13]
epi-Cubebol	11.42*	1491	[0.85]	11.63	1838	0.21
α -Selinene	11.42*	1491	[0.85]	9.56	1662	0.24
Bicyclogermacrene	11.42*	1491	[0.85]	9.68*	1672	0.79
α -Muuroolene	11.51	1498	0.69	9.68*	1672	[0.79]
β -Bisabolene	11.65	1509	2.54	9.81	1683	2.53
7-epi- α -Selinene	11.67*	1510	0.30	10.15*†	1711	[0.24]
Cubebol	11.67*	1510	[0.30]	12.19	1888	0.28
<i>trans</i> -Calamenene	11.78	1519	0.15	10.86	1771	0.16
δ -Cadinene	11.81	1521	1.89	10.07	1704	1.82
α -Calacorene	12.00	1536	0.06	11.74	1848	0.05
(<i>E</i>)- α -Bisabolene	12.08	1543	0.16	10.37	1729	0.15
Isocaryophyllene epoxide B	12.11	1545	0.16	11.76	1850	0.08
Germacrene B	12.18	1550	0.07	10.73	1760	0.07
(<i>E</i>)-Nerolidol	12.36	1564	0.16	13.45	2004	0.15
Spathulenol	12.44	1571	0.05	14.03	2060	0.07
Caryophyllene oxide	12.49*	1574	0.82	12.37	1905	0.66
Caryophyllene oxide isomer	12.49*	1574	[0.82]	12.30	1898	0.14
Unknown [m/z 161, 105 (84), 43 (80), 119 (72), 93 (62), 121 (54)... 204 (38), 222 (2)]	12.57	1581	0.01	13.62	2020	0.01
Humulene	12.68	1590	0.01	12.88	1952	0.01

epoxide I						
Humulene	12.81	1600	0.04	12.97	1960	0.04
epoxide II						
α -Corocalene	12.99	1614	0.02	13.30	1990	0.03
Guaia-6,10(14)-dien-4 β -ol	13.10*	1623	0.20	15.37	2193	0.16
Caryophylladienol I	13.10*	1623	[0.20]	15.69*	2226	0.05
Caryophylladienol II	13.16	1629	0.03	15.69*	2226	[0.05]
τ -Cadinol	13.26*	1637	0.09	14.55	2111	0.02
τ -Muurolol	13.26*	1637	[0.09]	14.69	2125	0.08
α -Muurolol	13.31	1641	0.28	14.83*	2139	[0.28]
<i>cis</i> -Calamenen-10-ol	13.44	1652	0.03	16.08	2267	0.04
<i>trans</i> -Calamenen-10-ol	13.56	1662	0.02	16.44	2305	0.03
(3 <i>Z</i>)-Caryophylla-3,8(13)-dien-5 β -ol	13.60	1665	0.02	16.51	2312	0.01
Unknown [m/z 43, 108 (62), 93 (51), 41 (42), 109 (37), 69 (36)...]	13.86	1687	0.01	16.38	2298	0.02
Total identified		98.37%			98.14%	
Total reported		98.54%			98.22%	

*: Two or more compounds are coeluting on this column

[xx]: Duplicate percentage due to coelutions, not taken into account in the consolidated total

†: Peaks apexes were resolved, but peaks overlapped and were summed for analysis

tr: The compound has been detected below 0.005% of total signal.

Note: no correction factor was applied

R.T.: Retention time (minutes)

R.I.: Retention index